Device quality Sb-based compound semiconductor surface: A comparative study of chemical cleaning

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We have studied the surface cleaning of Sb-based compound semiconductors using HF, NH_4OH, and HCl cleans and the metal–oxide–semiconductor (MOS) capacitors fabricated subsequently. GaSb, InGaSb, and AlGaSb surfaces are investigated using low-energy radiation from the synchrotron. Capacitance–voltage (*C–V*) and photoluminescence measurements are carried out on capacitors made with Al_2O_3 from atomic layer deposition and corroborated with the results from synchrotron spectroscopy. Excellent *C–V* characteristics with a mid-band-gap interface state density of $3 \times 10^{11}/cm^2eV$ are obtained on samples with the HCl clean. This is consistent with the finding that only the HCl acid clean is able to remove the native oxides present on GaSb and InGaSb surfaces, and produce clean and stable surfaces suitable for MOSFET development. Complete removal of AlO_x on the AlGaSb surface was not possible using chemical cleaning. Termination of AlGaSb with two monolayers of GaSb is proposed as a solution. © *2011 American Institute of Physics*. [doi:10.1063/1.3590167]

I. INTRODUCTION

Antimony (Sb)-based compound semiconductors have the highest electron and hole mobilities among all of the III–V materials. The electron saturation velocity in InSb is the highest among all the semiconductor materials. Room-temperature hole mobilities as high as 1500 cm^2/Vs in strained $In_{0.41}Ga_{0.59}Sb$ and GaSb channels at sheet charge densities near $10^{12}/cm^2$ have also been demonstrated recently.[1,7,8] The bandgap of Sb channels can be easily tuned from 0.18 eV for InSb to 0.72 eV for GaSb (direct bandgap) and 1.4 eV for AlSb (indirect bandgap). By combining these antimonide binaries in different stoichiometry, the bandgap can easily be tuned for detecting wavelengths in the 2–14 μm range. GaSb in particular has a bandgap that is well matched to the loss minima in optical fiber lines. In addition to a highly tunable bandgap, Sb-based channels offer high conduction and valence band offsets with lattice matched Al-containing Sb's making them suitable for confining carriers in thin 2D quantum wells for heterostructure-FET applications and lasing.

Schottky-gate FET devices with $In_xGa_{1-x}Sb$ channel have achieved an f_T of 305 GHz at 0.5 V V_{DS} ($L_G = 85$ nm) for *n*-channel[2] and an f_T of 140 GHz ($L_G = 40$ nm) for *p*-channel.[3] Although buried channel HEMT-like devices with excellent electron and hole transport[2,3] have been demonstrated, realization of an Sb-channel MOSFET has remained elusive due to the highly reactive nature of the Sb surface. This is illustrated in Fig. 1 where we show atomic force microscopy (AFM) scans of a nominally epiready GaSb surface for a sample removed from the package supplied by the vendor, which was sealed under nitrogen and measured immediately. The root mean square (rms) surface roughness is 0.73 nm. The rms roughness increases to 4.0 nm after exposure to atmosphere for a week. Thus, a stable and clean Sb surface suitable for dielectric deposition is the key if MOSFETs utilizing the high mobilities in these materials are to be realized. Surface cleaning and passivation is also important for infrared photodetectors made using Sb materials to improve the I_{ON}/I_{OFF} ratio and response time.

The cleaning of $InP/In_xGa_{1-x}As$ surfaces using various chemical cleans has been studied extensively,[4,5] which, in turn, has enabled MOSFETs utilizing the excellent transport properties in these materials. However, very little work has been reported on surface cleaning of Sb-based III–V semiconductors. In this paper we study the effectiveness of wet chemical cleans of HCl, HF, and NH_4OH, acids and bases used routinely in semiconductor processing, to remove the native oxides present on Sb surfaces and produce a stable and stoichiometric Sb surface suitable for device fabrication. Synchrotron radiation photoemission spectroscopy (SRPES) is used to study the top few monolayers of the surface after different chemical cleans and subsequent annealing in vacuum. Capacitance–voltage (*C–V*) and photoluminescence (PL) measurements are performed on capacitors fabricated after the various chemical cleans and correlated with the SRPES data.

Samples having the top surface terminated with GaSb, $In_xGa_{1-x}Sb$, or $Al_xGa_{1-x}Sb$ are studied to recommend the best material for surface termination. GaSb is an exciting material for developing a III–V *p*-MOSFET with high hole mobility. In addition, its bandgap is well suited for optical communication. $In_xGa_{1-x}Sb$ has both excellent electron and hole mobility and is considered as one of the candidates for replacing silicon in future technology nodes for transistors

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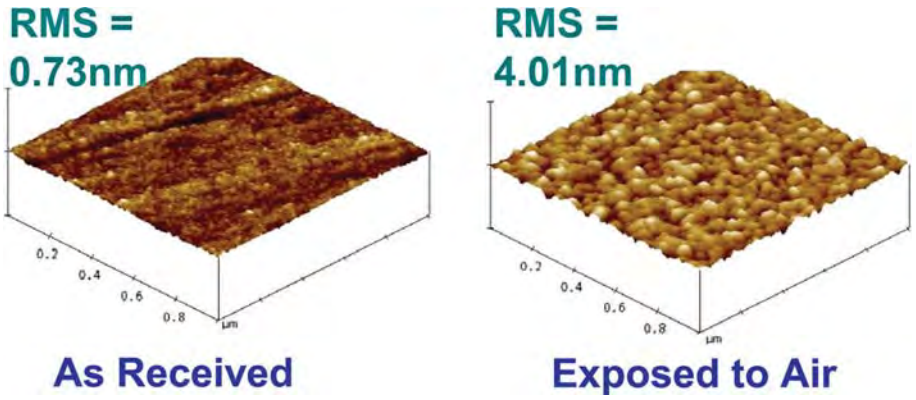

FIG. 1. (Color online) Tapping mode AFM map of the GaSb wafer: immediately taken out of a N_2 sealed package (left), exposed to air for a week (right). GaSb reacts with atmosphere rapidly to form thick native oxide on the surface.

where high performance and low power is required.[2] $Al_xGa_{1-x}Sb$ has been used extensively as a wide-bandgap barrier for buried channel $In_xGa_{1-x}Sb$ heterostructure FETs.

II. EXPERIMENT

A. Experimental details

The SRPES experiments were performed at beamlines 8-1 and 10-1 of the Stanford Synchrotron Radiation Lightsource. These two beamlines combined provide a tunable range of monochromatic photons from 60 to 1000 eV, yielding high surface sensitivity with a minimum electron escape depth around 5 Å. The Ga 3*d*, In 4*d*, Sb 3*d*, Al 2*p*, and the valence-band spectra can be studied with high accuracy in this low energy range.

The effectiveness of oxide removal by acid or base solutions was studied. We investigated three different chemicals, i.e., 9% HCl, 10% NH_4OH, and 2% HF, which are commonly used in semiconductor processing; the residual surface immediately after the chemical clean was studied. The chemical cleaning was done inside of a glovebox purged with pure argon; clean time was 2–3 min. After the cleaning the samples were rinsed in de-ionized (DI) water for 5 s and then they were blown dry by argon. We did not find any difference in photoemission spectra compared to samples without a DI water rinse. The glovebox was directly connected to the load lock of the photoemission chamber, allowing immediate transfer after the chemical cleaning without any exposure to air. In this manner the cleaning environment was controlled to minimize the contamination from air.

Last, the sample was vacuum annealed in the photoemission chamber. The sample surface–temperature to heater–current relationship was periodically calibrated by touching the surface of a test sample with a Chromel–Alumel thermocouple. The time for vacuum annealing was normally 30 min unless stated otherwise. In some figures the spectra collected were fitted with a Voigt function, which has a Gaussian broadened Lorentzian line shape. The deconvolution parameters are listed in Table I. The spin–orbit splitting value was taken to be 0.44 eV for Ga 3*d*, 0.90 eV for In 4*d*, 0.43 for Al 2*p*, and 1.25 eV for Sb 4d.[6] The branching ratio between the doublets was defined by the angular quantum number and was fixed at 1.5 for the *d*-orbital and 2 for the *p*-orbital The spectrum was collected at a 90° angle from the surface unless mentioned otherwise.

Al_2O_3 deposited by atomic layer deposition (ALD) was used for the capacitance–voltage characteristics study on GaSb surface. The sample preparation procedure before ALD deposition was the same as that used in SRPES study. The samples were loaded into the ALD chamber immediately after the chemical clean and the base pressure for the ALD chamber was 5×10^{-7} Torr. The deposition was done in a thermal ALD system at 300 °C using tetramethyl aluminum (TMA) and water as the precursors starting with a TMA pulse as the first step.

The surfaces we studied are listed in Table II. The GaSb (100) wafers used are *p*-type with carrier concentrations of 1–$2 \times 10^{17}/cm^3$ manufactured by Wafer Technology, UK. Samples having a top surface as $In_{0.20}Ga_{0.80}Sb$ and $Al_{0.7}Ga_{0.3}Sb$, were grown by MBE on GaAs (100) substrate using a metamorphic buffer.[7,8] We also studied the effect of chemical cleaning on the $Al_{0.70}Ga_{0.30}Sb$ sample terminated with the 2 monolayers (ML) of GaSb on the surface, and compare it with the sample having $Al_{0.70}Ga_{0.30}Sb$ termination. Thus, our sample space combined with different chemical cleans, formed a comprehensive study of chemical cleaning on Sb surfaces. This will serve as a useful reference for researchers working on GaSb MOSFETs, photodetectors, InGaSb surface-channel FETs, or utilizing AlGaSb as a barrier layer.

TABLE I. Photon electron spectroscopy parameters for In 4*d*, Ga 3*d*, Sb 4*d*, and Al 2*p* spectra.[a]

	Ga 3d	In 4d	Sb 4d	Al 2p
Spin–orbit splitting	0.44	0.90	1.25	0.43
Branching ratio	1.5	1.5	1.5	2
Gaussian width	0.45	0.42	0.34	0.23

[a]All energies are in electron volts.

TABLE II. List of Sb surface's studied and a look-up table for the associated SRPES figures.

Sample	Associated Figures
GaSb	1–4
$In_{0.20}Ga_{0.80}Sb$	7
$Al_{0.7}Ga_{0.3}Sb$	8 and 9
$Al_{0.7}Ga_{0.3}Sb$ with GaSb cap	8 and 9

B. Results on GaSb

Figure 2(a) plots the SRPES spectrum near Ga 3*d* and Sb 4*d* peaks for an as-received GaSb sample ($h\nu = 100$ eV). The *x*-axis in these plots is kinetic energy, thus the peak from the native oxides, i.e., GaO_x, SbO_x occurs on the left-hand side, i.e., at a lower kinetic energy (or higher binding energy) from the Ga 3*d*, Sb 4*d* peaks. The Ga 3*d* or Sb 4*d* peaks from the GaSb substrate are not visible at room temperature before any anneal and we observe only the peaks due to GaO_x and SbO_x present on the surface. When the sample is subsequently annealed in vacuum at 90, 200, 300, and 400 °C the SbO_x starts to desorb and the Sb 4*d* peak from the substrate below becomes visible. No desorption of GaO_x was observed even at 400 °C. The temperature of 400 °C was already quite high given the low melting point (705 °C) of GaSb.[9] Thus, the removal of native oxides was difficult by simple thermal annealing. We next explored the effect of HCl, NH_4OH, and HF clean in reducing the native oxides on the surface.

Figure 2 plots the Ga 3*d* and Sb 4*d* SRPES spectra after chemical cleaning in the (b) HF, (c) NH_4OH, and (d) HCl solutions and subsequent anneal in ultrahigh vacuum at 90, 200, 300, and 400 °C, using beamline 8-1 with photon energy of 90–100 eV. Use of low energy radiation from synchrotron results in increased surface sensitivity and allows observation of the spectrum from the top 2–3 ML of the surface with great accuracy. We observed that although the HF and NH_4OH cleans resulted in reduction of SbO_x on the surface and the Sb 4*d* from substrate peak became visible, the GaO_x on the surface was only partially reduced, using HF/NH_4OH clean. The GaO_x peak after HF/NH_4OH clean was only reduced slightly by subsequent anneal in vacuum, indicating that the residual GaO_x remaining after HF/NH_4OH cleaning cannot be completely desorbed with annealing in vacuum. Both the GaO_x and SbO_x appeared to be substantially reduced with the use of the HCl clean [Fig. 2(d)]. Further desorption of the remaining oxides was observed with thermal annealing and the surface appears to be completely free of native oxides after HCl clean and annealing at 300 °C. The peak separation between the SbO_x and Sb 4*d* peaks is ~2.90 eV, which was in between the known shifts of 2.76 and 3.56 eV for Sb_2O_5 and Sb_2O_3, respectively. Thus, GaO_x and SbO_x

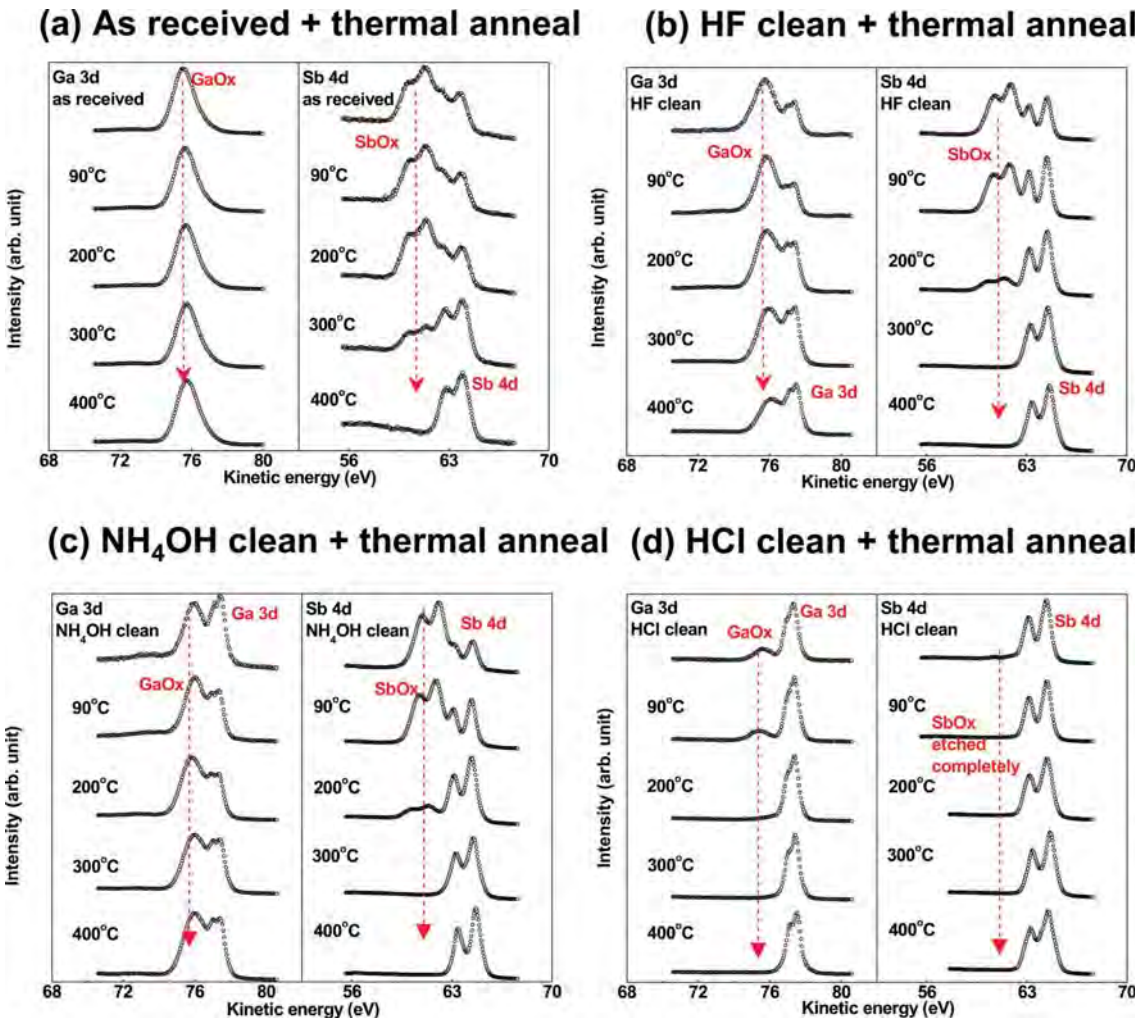

FIG. 2. (Color online) Low energy radiation from beamline 8-1 (photon energy = 90–100 eV) is used to study the effect of thermal anneal on (a) as-received sample and after chemical clean in (b) HF, (c) NH_4OH, and (d) HCl. Only the HCl acid clean is able to reduce both the GaO_x and SbO_x on the surface and yield a stable surface free of native oxide with subsequent thermal anneal.

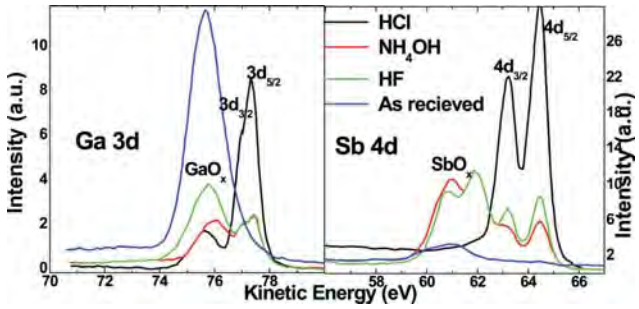

FIG. 3. (Color online) Synchrotron radiation from beamline 8-1 (photon energy = 90–100 eV) is used to observe the top 2–3 ML of the GaSb surface with high accuracy on (a) as-received sample; and after chemical clean in (b) HF, (c) NH_4OH, and (d) HCl.

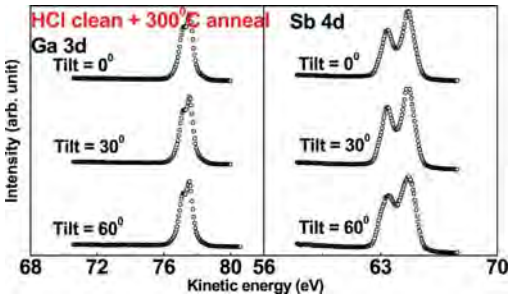

FIG. 4. (Color online) SRPES measurement at different angles (photon energy = 90–100 eV) is performed on the GaSb sample after HCl clean and 300°C anneal in vacuum to check for the presence of native oxides. No SbO_x or GaO_x is detected on the surface.

was used as a generic term for oxide peaks from Ga 3*d* and Sb 4*d*, respectively.

Figure 3 compares the spectrum from the GaSb surface immediately after the HCl, NH_4OH, and HF cleans. We observe that only the HCl-based clean is able to reduce both GaO_x and SbO_x on the surface. For the sample cleaned with HCl and then annealed at 300 °C (Fig. 4), measurements were performed at different tilt angles with respect to the surface to further increase the surface sensitivity. Again no SbO_x and GaO_x peaks were observed. Thus, we concluded from the SRPES study that the HCl clean of the GaSb surface and subsequent anneal results in a thermally stable GaSb surface free of native oxides. HF and NH_4OH cleans are unable to remove the GaO_x on the surface, which is not completely desorbed after subsequent thermal annealing.

C. GaSb passivation study using capacitance–voltage and photoluminescence measurements

Al_2O_3 was deposited at 300 °C by ALD using TMA and water as the precursors, after performing the chemical cleans with HCl, HF, and NH_4OH as discussed in the previous subsection. *C–V* characteristics were measured on capacitors on *p*-type GaSb having 70 cycles (~7 nm) Al_2O_3 dielectric and using a platinum metal gate. Figure 5 plots the *C–V* characteristics over a large frequency range from 1 kHz to 1 MHz. Nearly flat *C–V* characteristics were observed for the samples with no clean and HF clean. *C–V*'s on capacitors with the NH_4OH clean exhibited inversion response and a frequency dispersion in accumulation of 4%/decade, which was one indicator of high interface state density (D_{it}). The mid-bandgap D_{it} for the sample with HF clean was extracted to

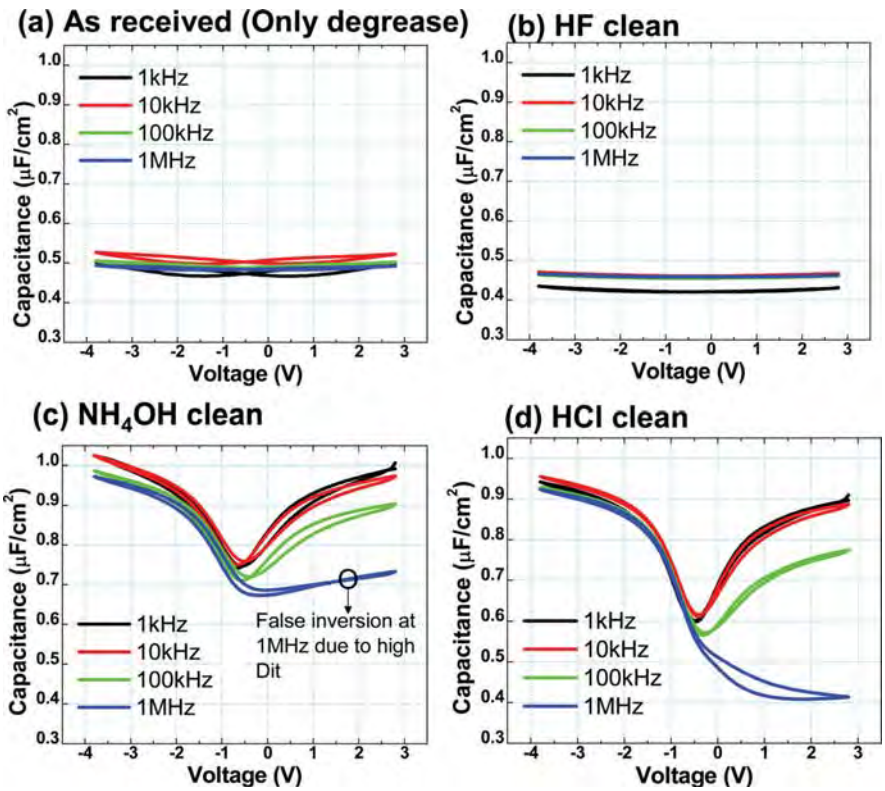

FIG. 5. (Color online) Bidirectional *C–V* characteristics are plotted from 1 kHz to 1 MHz for GaSb capacitors: (a) as-received; (b) HF clean; (c) NH_4OH clean; (d) HCl clean.

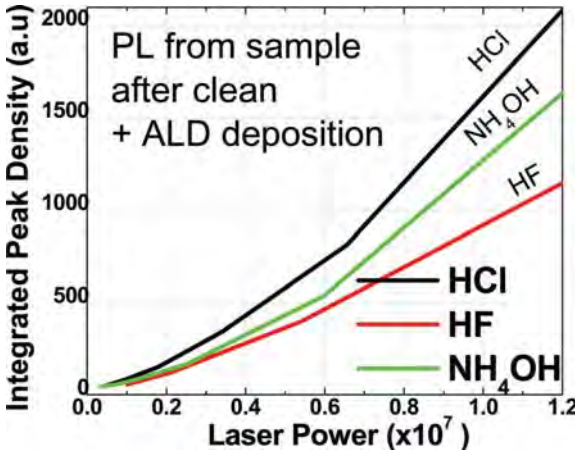

FIG. 6. (Color online) Integrated peak density of the photoluminescence (PL) signal is plotted as function of laser power. Higher PL signal intensity is observed in sample cleaned by HCl indicating better passivation.

be $5 \times 10^{12}/\text{cm}^2\text{eV}$ using the conduction method in depletion region.[10] The best *C–V* characteristics were observed for the sample with HCl clean, with the frequency dispersion in accumulation being less than 2%/decade. The mid-bandgap D_{it} for the sample with the HCl clean was extracted to be $3 \times 10^{11}/\text{cm}^2\text{eV}$ using the conduction method in the depletion region.[10]

PL has been proposed as a tool to compare the passivation achieved at the surface.[11] Figure 6 compares the integrated PL peak intensity for GaSb capacitors with the HCl, NH_4OH, and HF cleans. Increased PL intensity was observed in a GaSb sample with an HCl acid compared to samples with HF and NH_4OH clean, providing another independent proof for improved surface characteristics with HCl clean.

D. SPRES results on InGaSb, AlGaSb, and AlGaSb terminated with GaSb

So far we have discussed results on chemical cleaning and surface passivation of binary GaSb. In this section we discuss the surface cleaning of Sb ternaries, in particular $In_{0.20}Ga_{0.80}Sb$ and $Al_{0.70}Ga_{0.30}Sb$. Figure 7 plots the SRPES spectrum from the $In_{0.20}Ga_{0.80}Sb$ surface for the as-received sample and after NH_4OH and HCl cleans. Similar to the GaSb case, we observe that only the HCl clean is able to reduce the native oxides on the surface. The In 4*d* and Ga 3*d* spectrum are closely spaced and the In 4*d* peak is small due to the small relative concentration of indium (Fig. 7). The In 4*d*/Ga 3*d* spectrum are deconvolved [Fig. 7(b)] using the parameters listed in Table I. Figure 7(b) plots the spectrum after a HCl clean and a subsequent anneal in vacuum. The data can be completely fitted using the In 4*d* and Ga 3*d* peaks and no presence of native oxides is detected.

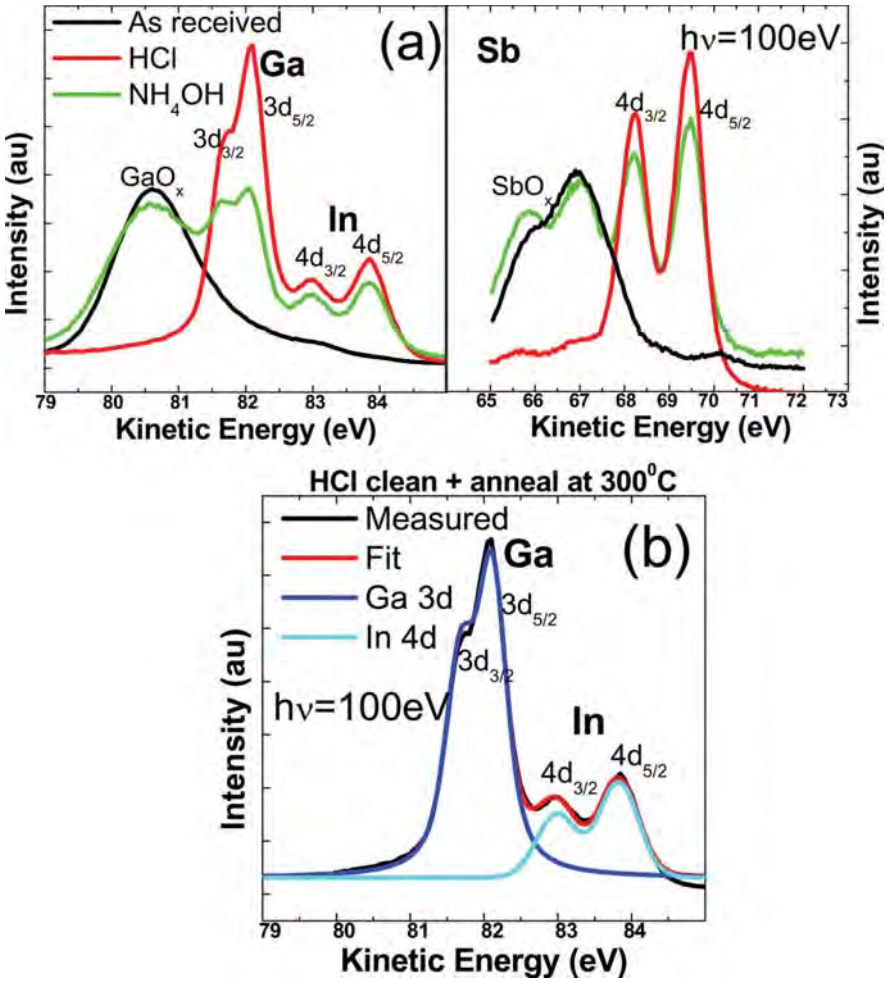

FIG. 7. (Color online) (a) In 4*d*, Ga 3*d*, and Sb 4*d* spectra are plotted for the $In_{0.20}Ga_{0.80}Sb$ surface for as-received sample and after NH_4OH and HCl clean. (b) The In 4*d* and Ga 3*d* spectrum are deconvoluted using parameters in Table I. No native oxide is detected on the surface after HCl clean and subsequent anneal at 300°C.

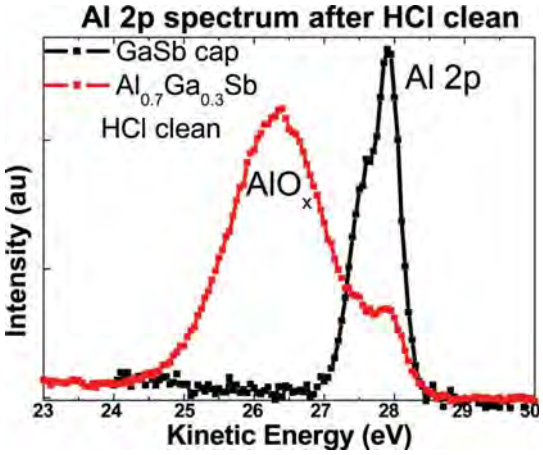

FIG. 8. (Color online) Al 2*p* spectrum after HCl acid clean is compared for the sample having a surface terminated with $Al_{0.70}Ga_{0.30}Sb$ and a sample having a 2 ML GaSb cap on top of $Al_{0.70}Ga_{0.30}Sb$.

Figure 8 plots the SRPES spectrum for Al 2*p* from the $Al_{0.70}Ga_{0.30}Sb$ sample with the HCl clean, which was found to be most effective in the removing the GaO_x and SbO_x oxide on the surface. The aluminum on the surface appears to be oxidized and only a small shoulder due to Al 2*p* from the substrate can be detected. The substrate Al 2*p* peak becomes more prominent after subsequent anneal in vacuum [Fig. 9(a)], but the AlO_x is not removed completely with annealing up to 400 °C. The removal of AlO_x was also not observed with the HCl and NH_4OH cleans (not shown). It must be noted that similar results have been observed in the case of Al-containing arsenides.[12] Kobayashi *et al.* recognized for the case of InAlAs that the AlO_x was hard to remove completely by typical wet cleaning, whereas the InO_x and AsO_x can be removed.[13]

Finally, we explore the use of 2 ML of GaSb (~0.6 nm) to terminate the $Al_{0.70}Ga_{0.30}Sb$ for which we found the AlO_x hard to remove using the chemical cleans. The GaO_x and SbO_x on the GaSb were removed using the HCl clean as discussed previously. The Al 2*p* peak from the $Al_{0.70}Ga_{0.30}Sb$ layer below could still be observed, as GaSb is only 2 ML thick. Figure 8 compares the Al 2*p* spectrum from the sample terminated with $Al_{0.70}Ga_{0.30}Sb$ and the GaSb terminated

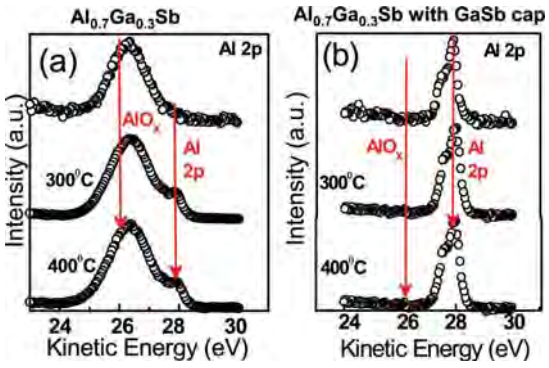

FIG. 9. (Color online) The effect of annealing after HCl clean on (a) $Al_{0.7}Ga_{0.3}Sb$ and (b) GaSb capped $Al_{0.7}Ga_{0.3}Sb$. The AlO_x is not desorbed with thermal anneal of the $Al_{0.7}Ga_{0.3}Sb$ sample (a); whereas the Al is not oxidized for the sample with a GaSb cap (b).

sample after HCl clean. No oxidation of the Al 2*p* peak is observed for the sample with GaSb termination. In Fig. 9(b) we observe that the Al peak remains stable and no oxidation is observed with subsequent anneals for the $Al_{0.70}Ga_{0.30}Sb$ sample terminated with 2 ML of GaSb.

III. CONCLUSION

The effect of HF, NH_4OH, and HCl chemical cleans on removing the native oxides and producing a stable surface with subsequent thermal annealing has been systematically studied using low-energy SRPES. We observe that only HCl is able to remove the native oxides on the InGaSb and GaSb surface and subsequent thermal annealing yields a stable surface suitable for dielectric deposition. The results from the SRPES study correlate well with the *C–V* and PL measurements. Excellent *C–V* characteristics with frequency dispersion of less than 2%/decade and a mid-bandgap D_{it} of $3 \times 10^{11}/cm^2eV$ are demonstrated on GaSb substrates after an HCl clean.

The removal of the AlO_x on the AlGaSb surface was not possible using a chemical clean. Termination of AlGaSb with GaSb is proposed as a solution. A stable surface with no oxidation of Al can be obtained for an AlGaSb terminated with just 2 ML (~0.6 nm) of GaSb. This is an encouraging result as the thickness of GaSb layer is very thin to avoid any parallel conduction when a high-*k* dielectric such as Al_2O_3 is subsequently deposited on such a surface for heterostructure MOSFET fabrication.

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